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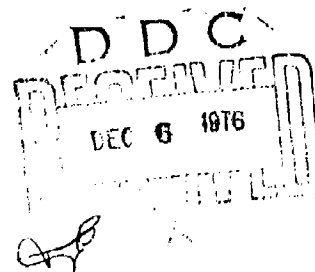
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GRAIN BOUNDARY SEGREGATION AND  
STRESS CORROSION CRACKING OF  
ALUMINUM ALLOYS

by  
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SECTION STATEMENT A

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CRACKING OF ALUMINUM ALLOYS

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## ABSTRACT

Auger electron spectroscopy and chemical depth profiling by argon sputtering were employed to obtain the grain boundary segregation profiles of various aluminum alloys. Samples of both commercial (7075, 7050, and 7049) and high purity alloys based on the Al-Zn-Mg ternary in different heat treatments were examined following in situ fracturing in the spectrometer. Segregation profiles indicate that the grain boundaries in these alloys are considerably enriched in Mg and Zn. In commercial alloys, however, the grain boundaries are depleted in the minor elements Fe, Cu, and Si. AES spectra of oxide films formed on Al-Zn-Mg alloys indicate that the enhanced segregation along the grain boundaries results in a film rich in Mg. It is postulated that incorporation of extensive amounts of these alloying elements into the film renders it less protective and leads to enhanced hydrogen entry and embrittlement by stress corrosion cracking.

## I. INTRODUCTION

Mechanistic studies carried out during the past few years have helped elucidate both the microstructural features<sup>(1-4)</sup> and environmental factors<sup>(5-8)</sup> associated with the propagation of stress corrosion cracks in an Al-Zn-Mg ternary alloy. These studies led to the view that the rate-controlling step in crack propagation, within the precipitate-free zone (PFZ) of the ternary alloy, was the stress-assisted dissolution of the aluminum solid solution interspersed between the MgZn<sub>2</sub> particles at the

grain boundary. Thus, a non-susceptible structure in these alloys was identified as one containing a large spacing between  $MgZn_2$  particles.

Recently, however, the conclusion that the SCC of high-strength aluminum alloys occurs by some form of electrochemical dissolution has been challenged. The investigators<sup>(9, 11)</sup> who question this view suggest instead that cracking is due to some form of hydrogen embrittlement. Specifically, Swann and Montgrain<sup>(9)</sup> cite experiments carried out within a high-voltage electron microscope in which an Al-Zn-Mg ternary suffered embrittlement in moist air. In this experiment, the  $MgZn_2$  precipitates apparently were not dissolved and the crack passed along the incoherent interface between the grain boundary precipitates and one of the grains. Although these results may be criticized in that moist air does not represent a realistic stress-corrosion environment, the conclusions have been supported by both Spiedel<sup>(10)</sup> and Gest and Troiano<sup>(11)</sup>.

In an attempt to distinguish between these two models, the influence of loading mode on the stress corrosion susceptibility was examined using a commercial 7075-T6 alloy. Here, the rationale was that the observation of different susceptibilities under different loading modes would provide evidence to support a model of cracking involving the stress-assisted diffusion of hydrogen to interact with a crack tip<sup>(12)</sup>. Conversely, if cracking requires the breakdown of surface films and/or extensive dissolution then there should be less, if any, dependence of failure on loading mode.

The conclusion of this investigation<sup>(13)</sup> was that in fact both dissolution and hydrogen embrittlement mechanisms operate to promote cracking. Of these, the hydrogen mechanism is dominant, while the dissolution process serves primarily to supply the source of hydrogen. Recent experiments by Scamans et al.<sup>(14)</sup> on the pre-exposure embrittlement of Al-Zn-Mg alloys tend to reinforce this conclusion.

Accepting this result, critical issues now relate to the interaction of hydrogen with the detailed microstructure of the grain boundary region. To understand this interaction, Auger electron spectroscopy (AES) and depth profiling by argon sputtering have been employed to obtain detailed information on the composition immediately adjacent to the grain boundary.

A limited number of attempts have been made to measure grain boundary segregation in binary and ternary Al-Mg alloys by other investigators. For instance, Cundy et al.<sup>(15)</sup> demonstrated local Mg enrichment within 500 Å of the grain boundary in a quenched binary Al-7 wt % Mg alloy using the technique of combined electron microscopy and electron energy analysis. Using a similar technique, Doig and Edington<sup>(16)</sup> obtained composition profiles of Mg across grain boundaries in a quenched Al-Mg-Zn alloy. These composition profiles indicated that the grain boundaries in the brine-quenched samples were enriched in Mg while the oil-quenched samples containing fine precipitate along the boundaries were depleted in Mg. Although the electron microscopy-microanalysis (EMMA) technique has a spatial resolution of 100 Å, the resolution in composition is only 0.5 wt %. Also, the compositional profiles were

obtained on thinned samples (to make them transparent to the electron beam) and may not be representative of bulk samples.

Shastri and Judd<sup>(17)</sup> employed electron probe microanalysis to obtain segregation profiles on grain boundaries in Al-Zn-Mg alloys. The extent of segregation was large ( $\sim 7 \mu\text{m}$ ) and the amount of segregation rather small. Probably, these results are in error due to limitations in spatial resolution (a few microns at the most) and in composition determination. Differences in composition between the grain boundary and grain interior were small and subject to large errors due to instrumental drift and background intensity variations. Further, the analysis was performed only on the surface and not on fractured samples.

The technique of Auger electron spectroscopy, on the other hand, is very surface sensitive and the compositional information comes only from the first few atomic layers. When combined with  $\text{Ar}^+$  ion sputtering it becomes a powerful tool with good spatial and compositional resolution. One drawback, however, is that the segregation profiles are obtained from several grains and local variations are averaged. In spite of this, AES analysis provides valuable information on chemical composition differences between the grain boundary and the interior. We have employed this technique to obtain segregation profiles in both high purity Al-Zn-Mg ternary and commercial alloys. The results of these experiments and their significance to stress corrosion cracking in high strength Al-alloys are described in this paper.

## II. EXPERIMENTAL METHODS AND MATERIALS

The instrument used for the AES study is a Physical Electronics Model 548 Auger/ESCA spectrometer, which features a double-pass cylindrical mirror analyzer and an ultra high vacuum system (UHIV). The Auger signal is excited by a 5 KeV primary electron beam from an electron gun housed coaxially inside the analyzer. Most AES measurements are made with a focussed beam of 200  $\mu\text{m}$  diameter and 50 to 60  $\mu\text{A}$  current. The system is equipped with an  $\text{Ar}^+$  ion sputtering gun, capable of producing 2 KeV ions. All the experiments were conducted under a basal pressure of less than  $5 \times 10^{-10}$  torr, except during ion sputtering. During sputtering, the UHIV system is filled with pure argon to a pressure of  $5 \times 10^{-5}$  torr, while liquid nitrogen is used to cool the lower section of the UHIV system via a cryo-design to further trap oxygen and other impurity gases. The system has a multiplex unit, which enables one to plot the peak-to-peak height of six different Auger peaks consecutively, and is indispensable for monitoring depth profiles. A fracture device, utilizing a shear-to-break configuration was used for in situ fracture experiments.

To obtain quantitative information from the depth profiles, the peak-to-peak heights of the elements were expressed as a ratio of the peak-to-peak height of the matrix element Al(1390 eV). Conversion from such ratios to approximate atomic percentages requires the use of elemental sensitivities. The peak-to-peak height ratios were then normalized by using the sensitivity factors listed in the "Handbook for Auger

Electron Spectroscopy" by Physical Electronics Industries (PEI), Inc. (18) We realize that there are pitfalls associated with this comparatively simple approach especially if the elements are not distributed uniformly within the sampling depth. The typical sampling depths at the most prominent Auger peaks for the two elements of interest, Mg and Zn are several atomic layers and as long as the segregation is not confined to only one monolayer such a procedure is reasonable. Another source of error is that the sensitivity factors listed in the Handbook were obtained from either pure elements or compounds and not from solid solutions. Thus, we point out that although this procedure will not alter the qualitative trends in the depth profiles the exact values at any given depth may be slightly in error.

The materials used for the AES studies were a high purity Al-Zn-Mg ternary alloy in various heat treated conditions, and the high strength alloys, 7075, 7050, and 7049. The high purity ternary used for these investigations had a nominal composition of 5.5 wt % Zn and 2.5 % Mg. Strips 1 mm thick were solution treated in dry argon at a temperature of 475°C for 10 mins. and 2 hrs. and water quenched. The grain sizes for the two solution treatments were 0.08 and 0.25 mm respectively. The quenched samples were then heat treated at 130°C and 160°C for 0, 4½, and 22 hrs. to obtain the under-, peak-, and over-aged conditions. Small strips were cut from the samples, notched and transferred to the spectrometer. In spite of repeated attempts, the large grain size samples could not be fractured in the spectrometer. Hence, grain boundary concentration profiles could only be obtained on the small grain size samples.

AES studies were also conducted on the commercial alloys in different tempers. Specifically 7075, 7049, and 7050 Al-alloys in T73 and T76 tempers were employed. The detailed chemical composition of the alloys is listed in Table I.

### III. RESULTS

#### 1. High Purity Ternary Alloy:

A scanning electron micrograph of the fracture surface of the fine-grained ternary is shown in Fig. 1. Fracture is clearly intergranular. The electron beam used for the AES measurements is roughly 200  $\mu$  in size and under these conditions an average of 6 to 8 grains will be analyzed. The fracture surface also has a macroscopic roughness which might influence the AES measurements and the chemical depth profiles to some extent. However, there is no easy way to take this into account.

A typical Auger spectrum taken from the fracture surface of the ternary alloy, quenched and aged at 160° C for 4½ hrs. is shown in Fig. 2. The spectrum indicates the presence of the major alloy additions, Mg and Zn. The grain boundary depth profiles obtained on the ternary alloy in different heat treated conditions are shown in Figs. 3 and 4. The age-hardening curves at these two temperatures are superimposed on the profiles. Under all heat treatment conditions there is marked segregation



of Zn and Mg to the grain boundaries. The extent of segregation is roughly 250 to 500 Å and the grain boundary concentrations are several times the bulk concentrations. These results are in agreement with some of the results obtained by Cundy et al.<sup>(15)</sup> and Doig and Edington<sup>(16)</sup>, but completely at variance with the results of Shastry et al.<sup>(17)</sup>. Shastry and Judd observed solute segregation to extend as far as 7 µm from the boundary. This discrepancy is most probably due to spatial and compositional resolution limitations of the microprobe employed and the fact that the analysis was carried out only on the alloy surface.

Although the depth profiles are sensitive to heat treatment, the changes are rather small. Also, the electron beam averages information over an area several grains in diameter and local changes in the segregation profile may well be obscured. The relatively minor changes in the overall depth profiles as a function of heat treatment may include rather large changes in local segregation. In spite of this, certain features of the chemical depth profiles provide valuable information.

For instance, the depth profiles also include information from the MgZn<sub>2</sub> particles on the grain boundaries. Remembering that the depth profiles are normalized to an Al peak-to-peak height of 100, the amount of Mg should only be about one half of that of Zn if the segregation profiles were exclusively due to contributions from the second phase particles. This, however, is not the case. In almost all cases the grain boundaries contain excess Mg. The significance of this observation and its relevance to stress corrosion cracking will be discussed later.

## 2. Commercial Alloys:

A typical Auger spectrum from a 7075 T73 alloy is shown in Fig. 5. In addition to the principal alloying additions, Mg and Zn, small amounts of Fe, Si, and Cu are present. The normalized depth profiles for 7075 in two different tempers, T73 and T76, are shown in Fig. 6. An interesting observation is the difference in the segregation patterns of the minor and major alloying additions. The grain boundaries, while enriched in the major alloying additions, Mg and Zn are depleted in Fe, Cu, and Si. The minor elements show an inverse segregation profile. This difference in the segregation profiles was observed in all commercial 7000-series alloys.

Fig. 7 is an Auger spectrum from a 7050-T73 alloy. Notice the large amount of Pb present in the spectrum. Pb is a common impurity in Zn and is present in most commercial alloys. However, the large amounts of Pb observed in both 7050 and 7049 alloys were not observed in the 7075 alloys. The reasons for this difference appear to be related to production practices at the mill. For example, most heats of the newer alloys are made from pure components or master alloys, whereas 7075 is generally prepared with some proportions of scrap.

Chemical depth profiles for the 7050-T73 and 7050-T76 alloys are presented in Fig. 8. The grain boundaries are enriched in Mg, Zn, and Pb and depleted in Fe, Cu, and Si. Qualitatively the profiles are similar to those observed in other commercial alloys except for the large amounts of Pb present at the grain boundaries.

AES measurements were also made on the 7049-T73 and 7049-T76 alloys and the results are shown in Fig. 9. Once again the composition profiles are very similar to those observed in the 7050 alloys. The grain boundaries are enriched in Mg and Zn and depleted in Fe, Si, and Cu.

#### IV. DISCUSSION AND CONCLUSIONS

Stress corrosion cracking is the result of a complex interplay between the alloy and the environment. Recent evidence<sup>(9,11,13)</sup> seems to indicate that hydrogen embrittlement is one, and perhaps the dominant, of the mechanisms in stress-corrosion cracking. Accepting this evidence, it is essential to consider the following:

- i) Generation of hydrogen
- ii) Entry of hydrogen through the protective surface film
- and iii) Concentration of hydrogen in a localized region to cause embrittlement.

Each of these critical steps is governed by the interaction between the bulk microstructure, the grain boundary microstructure and the environment. For example, the ability of the alloy to concentrate hydrogen in a local region for a given loading mode is enhanced by higher yield strengths and planar slip modes. This would explain why alloys in the peak-aged condition are most susceptible. Entry of hydrogen in the proximity of a grain boundary is controlled by the oxide film which, in turn, is determined by the chemical composition adjacent to the grain boundaries. The generation of hydrogen is also controlled by the interaction between the environment and the grain boundary microstructure.

The AES studies have yielded an improved picture of the segregation effects at the grain boundaries. The qualitative aspects of the segregation patterns are quite similar in many of the commercial alloys studied so far. It might be argued that it is difficult to reconcile relatively small changes in segregation with large differences in susceptibility<sup>(19)</sup>. Clearly, segregation is only one of the many variables in the complex material/environment interplay leading to stress corrosion cracking and cannot explain the complete stress-corrosion cracking behavior. Yet, these studies have indicated extensive segregation of Mg and Zn to the grain boundaries in all cases. Segregation influences not only the local electrochemistry of the grain boundary region but also the nature of the oxide film. Since pure aluminum oxide is a very effective barrier against hydrogen dissociation and entry<sup>(20)</sup>, it follows that incorporation of the various alloying additions that are segregated near the grain boundary into the film will probably reduce its protective nature. Clearly, extensive incorporation of Mg into the film would result in a less protective film since magnesium is considerably more soluble than aluminum over neutral and the mildly acidic pH ranges<sup>(21)</sup> typical of that present at propagating crack tips. This reduces its ability to serve as an effective barrier against hydrogen dissociation and entry, as illustrated schematically in Fig. 10.

To establish the validity of these ideas some preliminary results obtained on the composition of the oxide films formed on the high purity

ternary alloy are presented. The as-received sample was heat treated at 475°C for 10 minutes in dry argon, water quenched and transferred immediately into the spectrometer. A typical AES spectrum is shown in Fig. 11. Notice the extremely large amount of Mg present in the oxide. The Mg to Al peak-to-peak height ratio is 3.64 -- in other words, the Mg content of the oxide exceeds that of Al. On the other hand, the Zn to Al peak-to-peak height ratio is only about .048. Although the atomic ratios of Mg and Zn are roughly the same, the amount of Mg in the oxide film is disproportionately high.

Further studies now underway suggest that the Mg content of the films is sensitive to heat treatment, grain size, storage conditions, and the relative humidity of the environment. In addition to changes in the average Mg content of the film there are large local variations in a given sample. These local variations seem to fall into two types of areas, Mg rich and Al rich, and seem to have a definite correlation with the grain size. This would be expected if the segregation of Mg induces large changes in the local composition of the film.

To summarize, AES studies show that the alloying elements Mg and Zn are heavily segregated along the grain boundaries. This segregation induces dramatic changes in the composition of the film. For equal atomic concentration Mg plays a more dominant role than Zn. These composition changes are sensitive to alloy and environment variables. It is postulated that a film locally enriched in Mg, particularly along the grain boundaries will be less effective as a barrier against hydrogen dissociation and entry and enhances hydrogen embrittlement and stress corrosion cracking. Such enrichment would be critical to both the initiation and propagation of SCC cracks.

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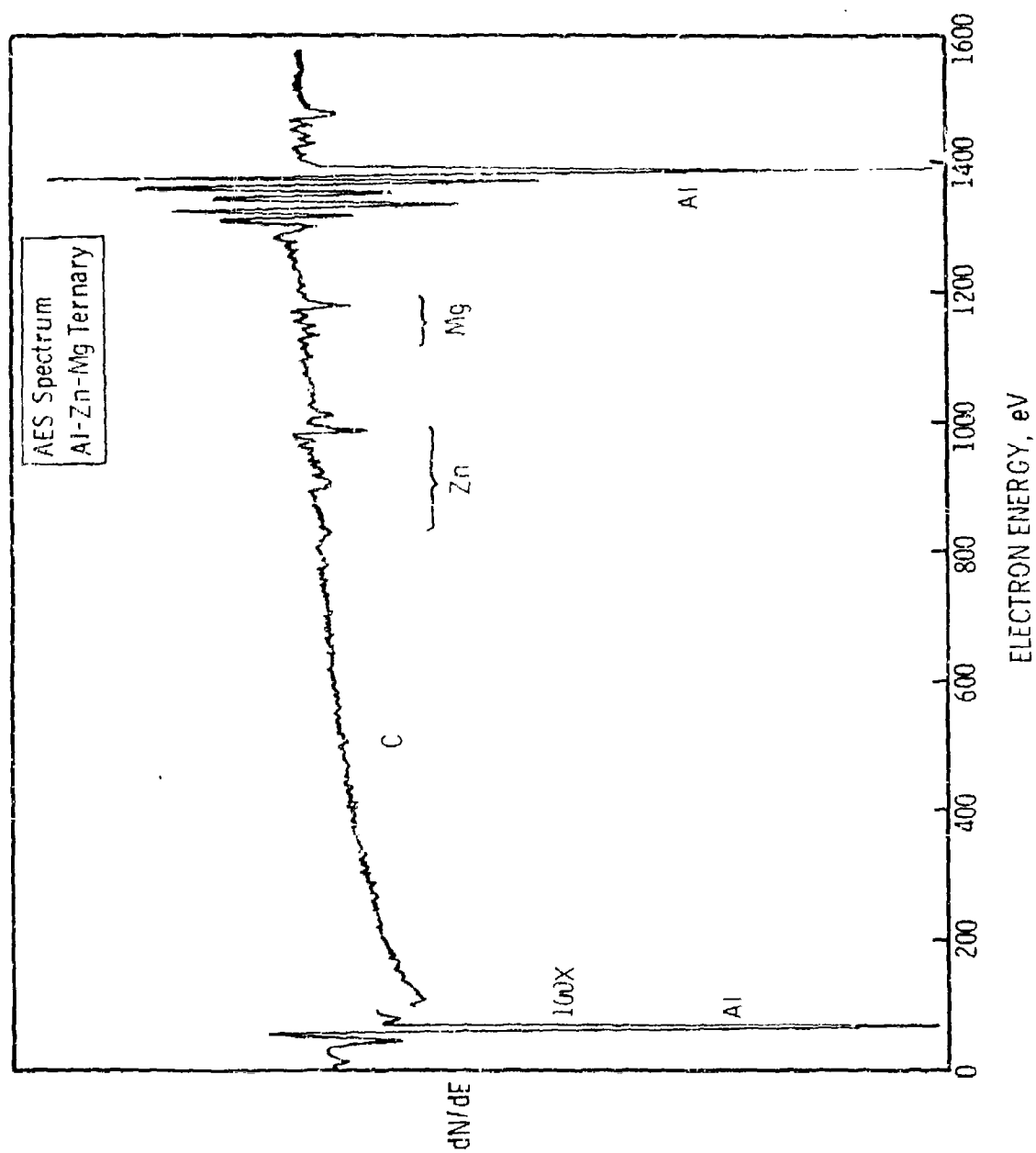
TABLE 1.

Detailed chemical composition in weight percent of the 7000 series alloys used for the AES measurements.

Alloy	7075	7050	7049
Si	0.08	.05	0.03
Fe	0.11	.10	0.09
Cu	1.4	2.3	1.5
Mg	2.4	2.4	2.49
Zn	6.05	6.35	7.75
Pb	0.00	0.02	0.04
Cr	0.20	0.03	0.21
Zr	0.00	0.12	0.00
Mn	0.01	0.01	0.01

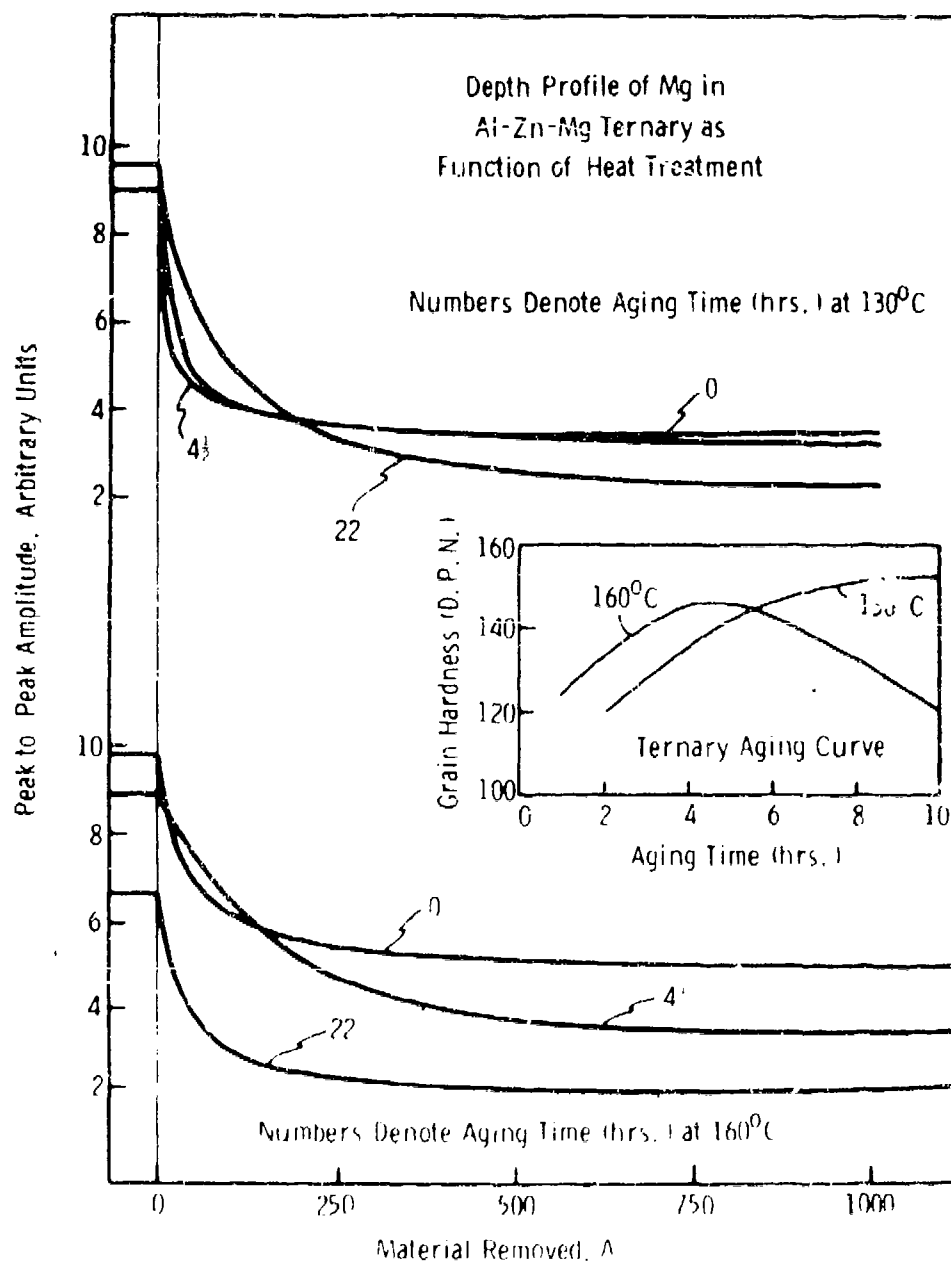


1. Scanning electron micrograph of the fracture surface of a quenched and aged high purity Al-Zn-Mg ternary alloy. The grain size is approximately 0.08 mm (x100).

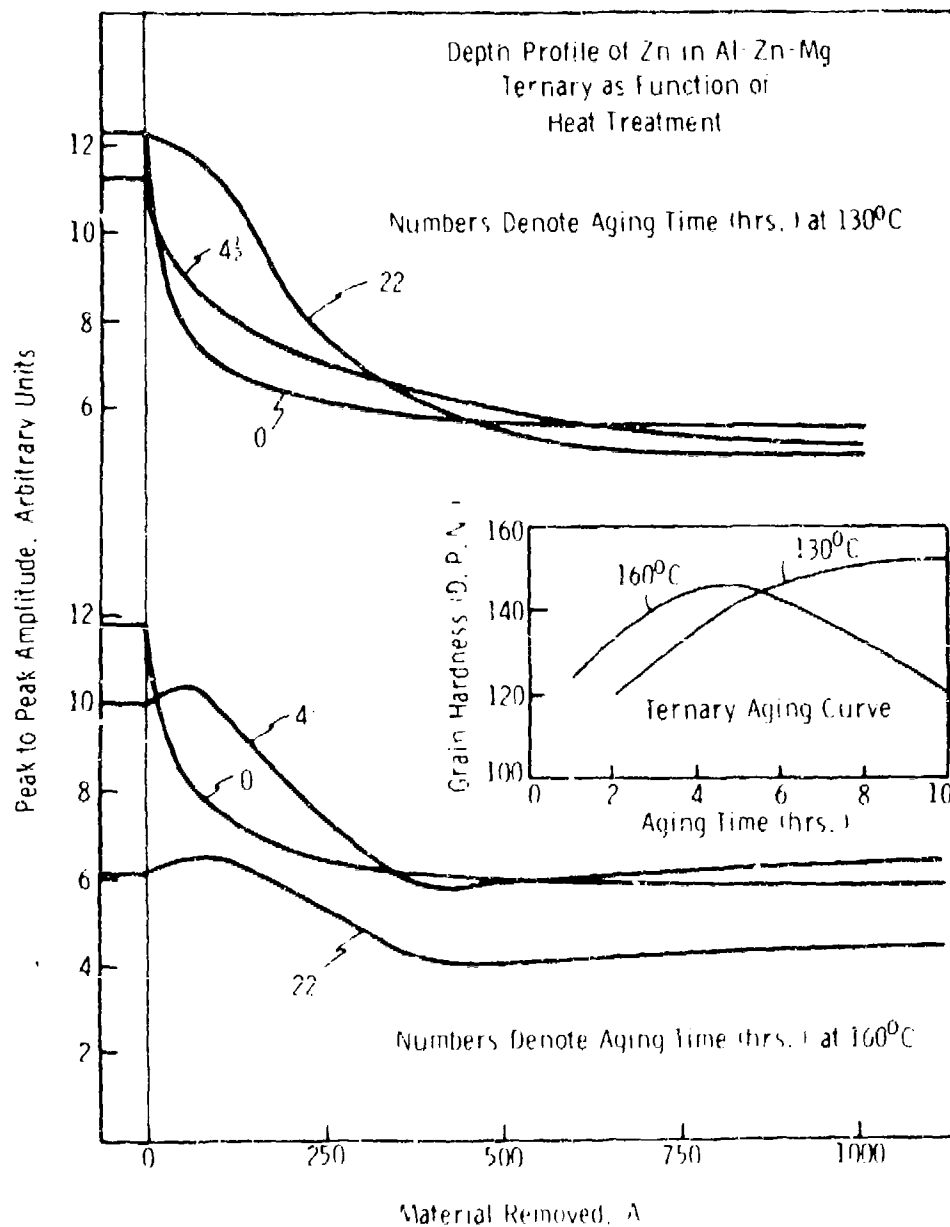


2. Typical Auger electron spectrum from the fracture surface of a quenched and aged high purity ternary alloy.

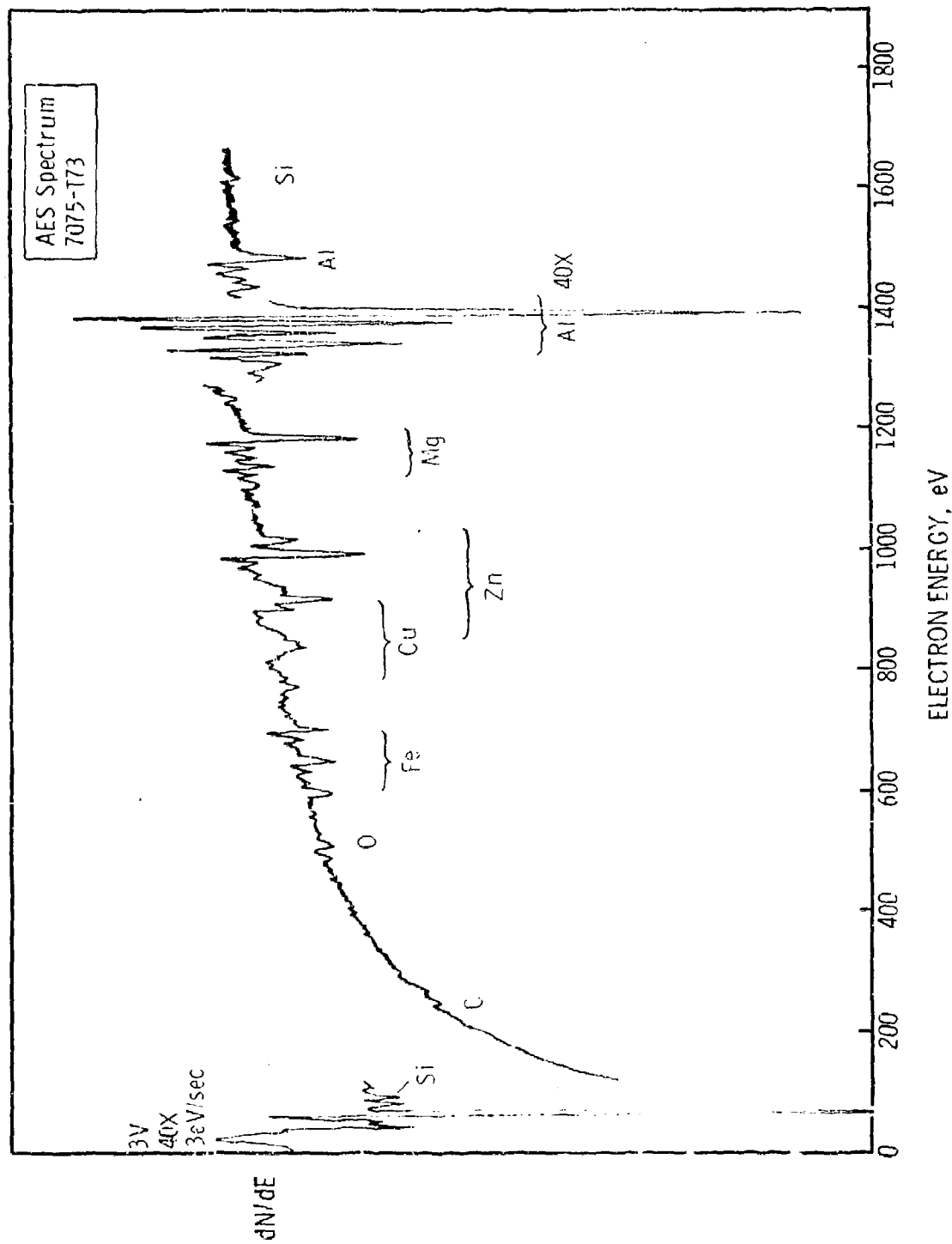




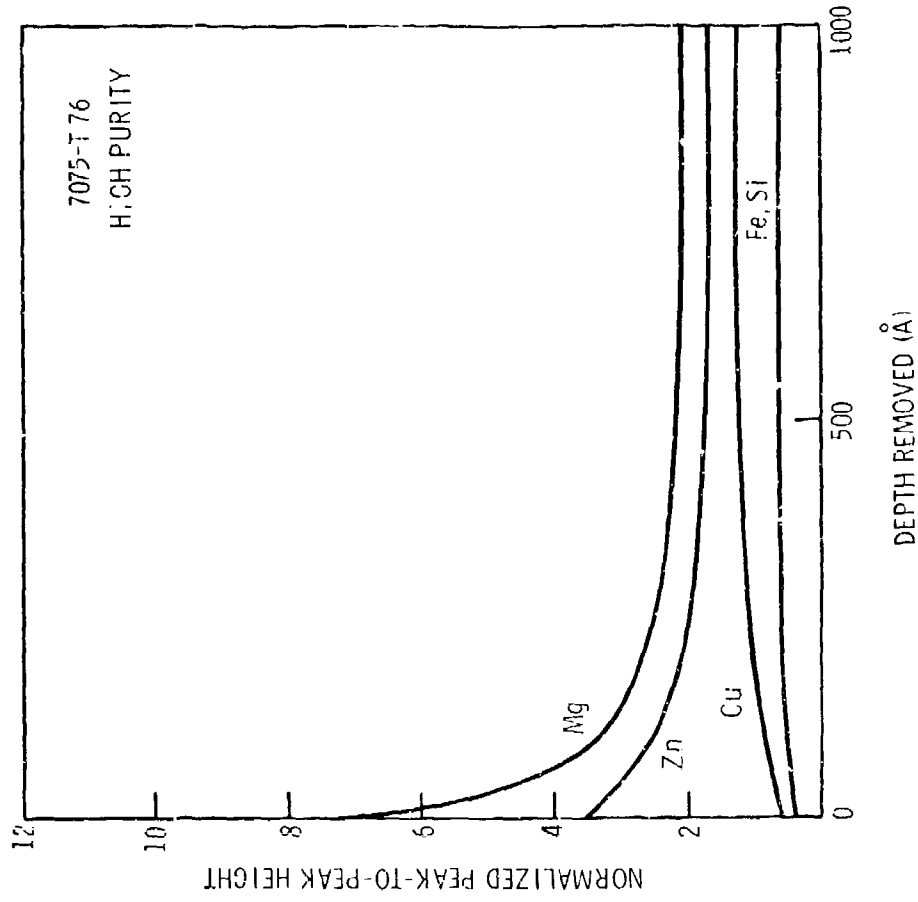
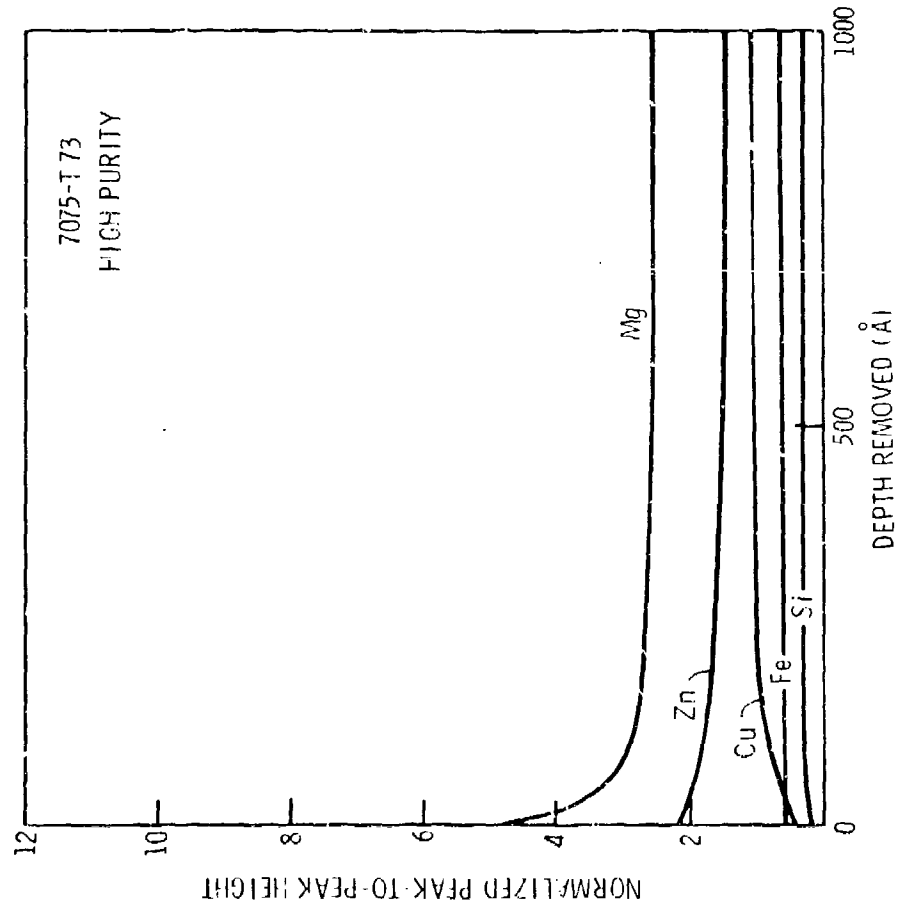
3. Chemical depth profiles for Mg in Al-Zn-Mg ternary alloys as a function of heat treatment. The aging curves at 130°C and 160°C are superimposed.



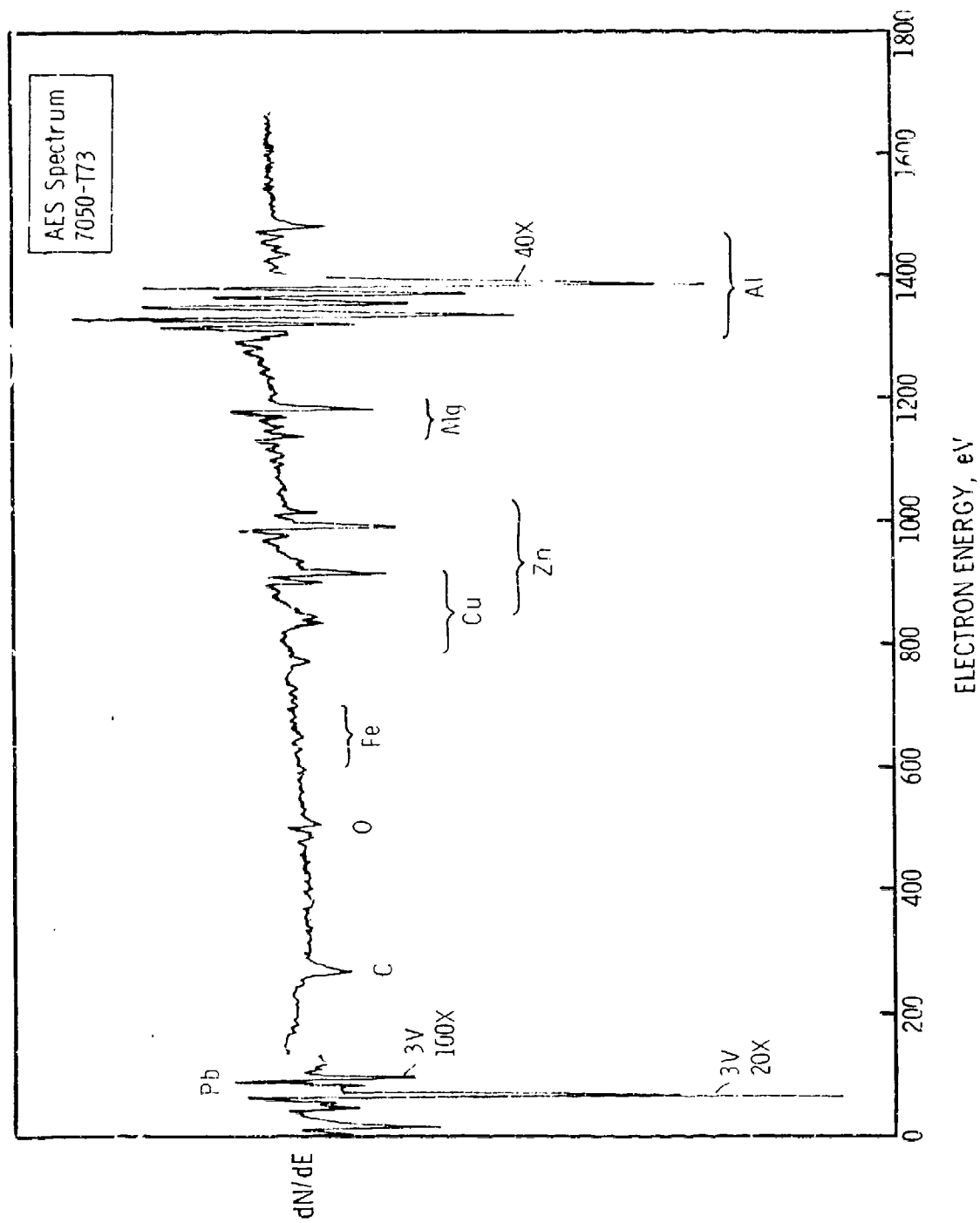
4. Chemical depth profiles for Zn in Al-Zn-Mg alloys as a function of heat treatment. The aging curves are superimposed.



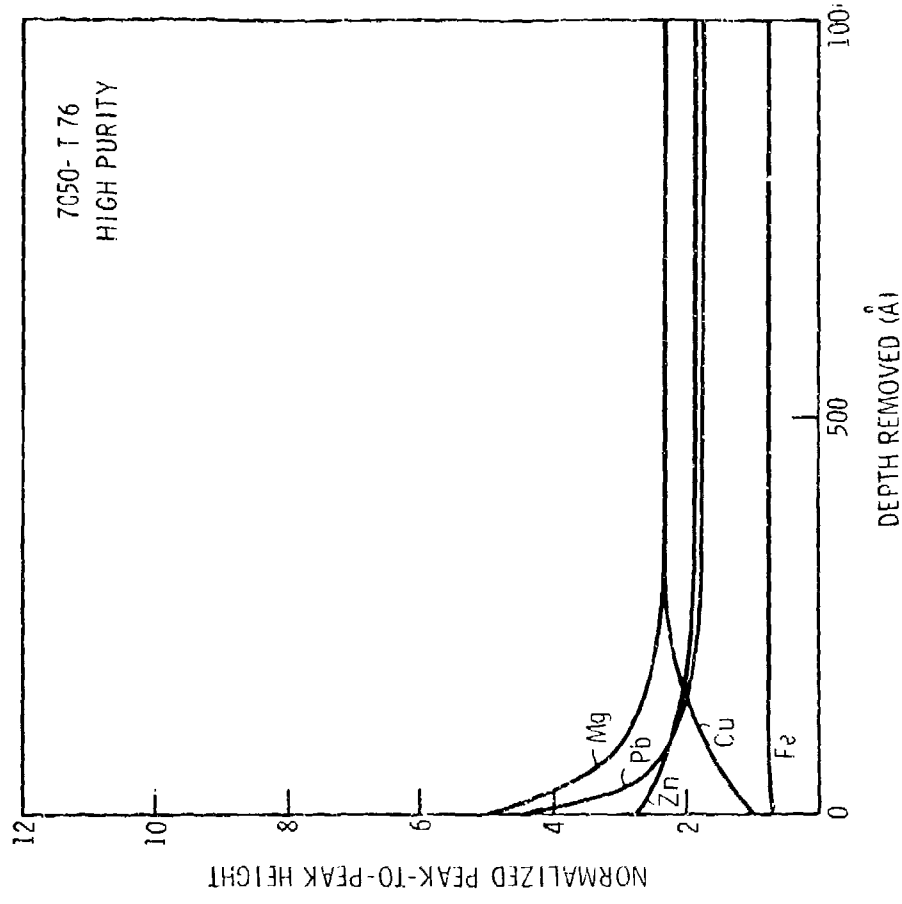
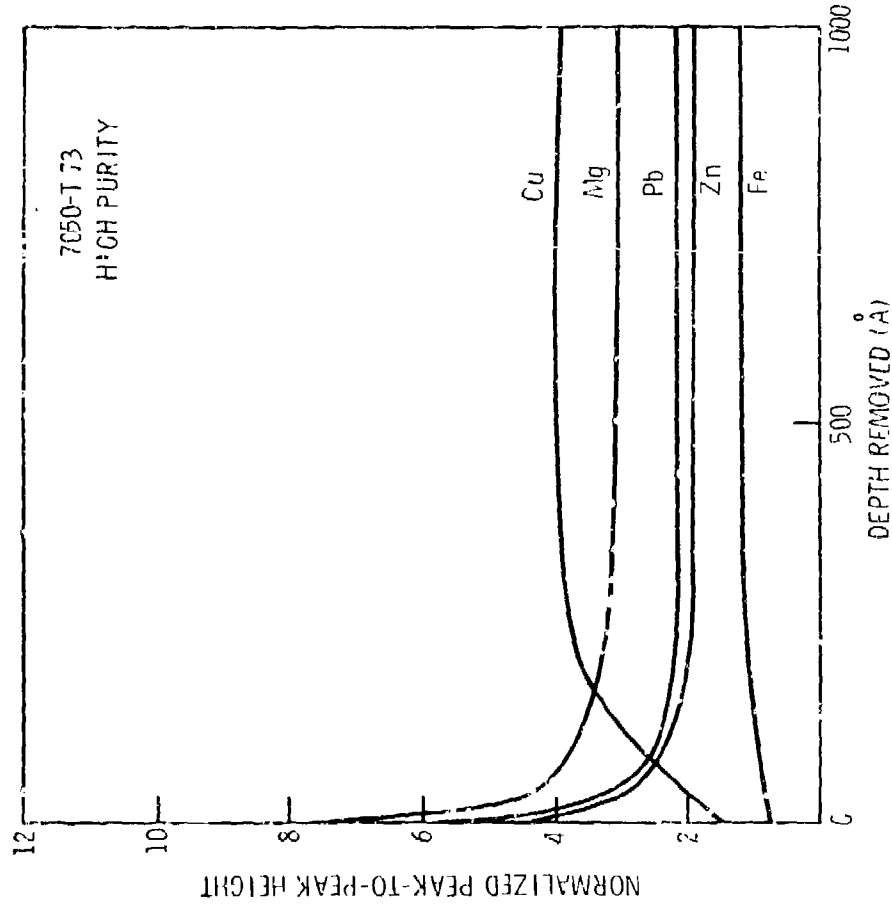
5. A typical Auger spectrum from the fracture surface of a 7075-T73 Al alloy.



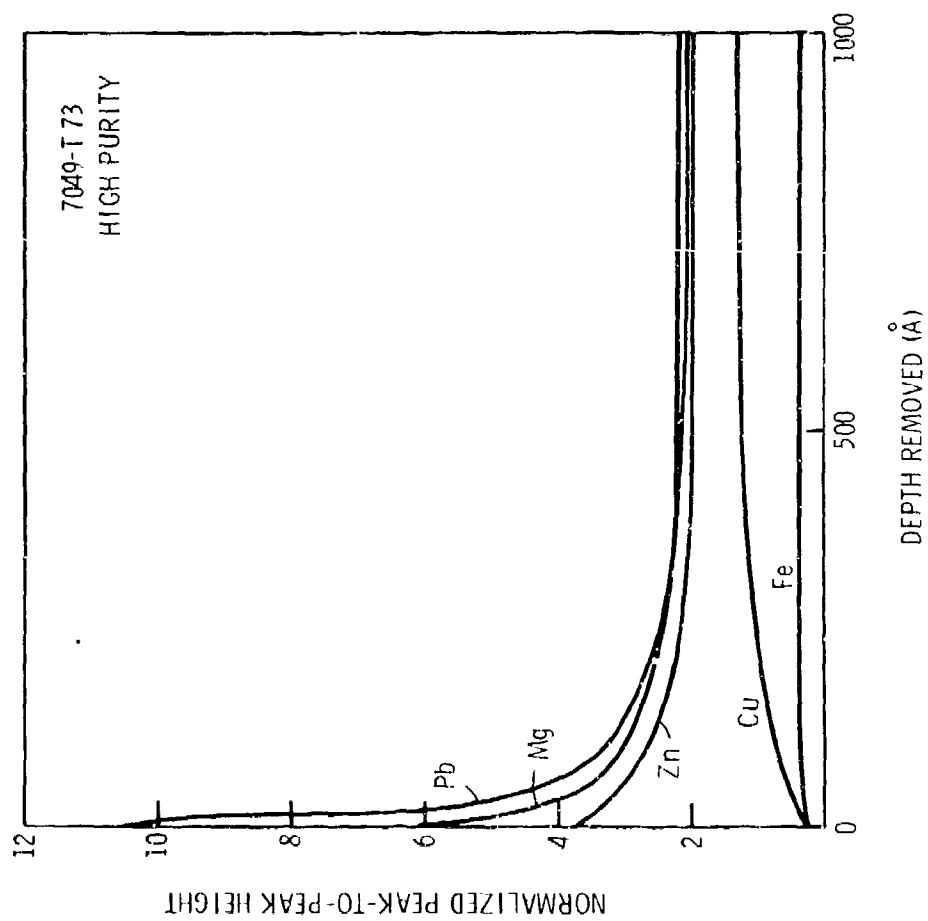
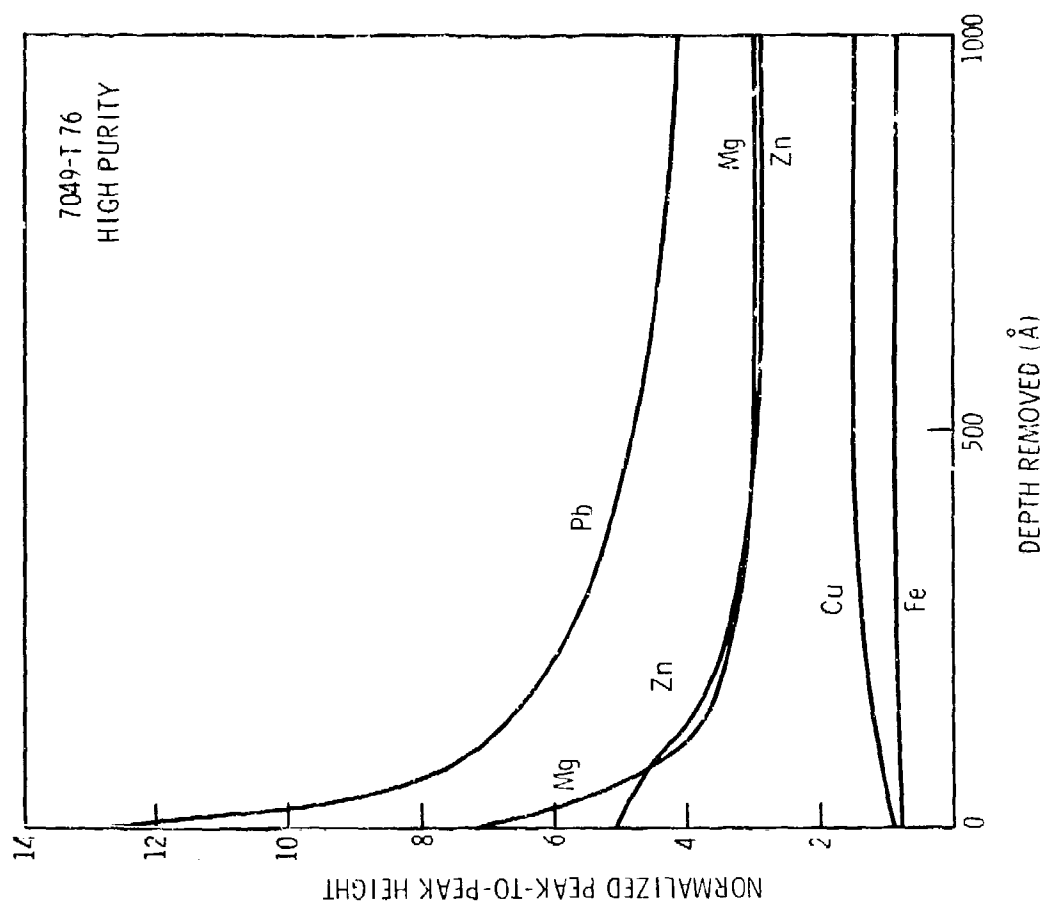
6. Chemical depth profiles of 7075 Al alloy in two tempers, T73 and T76.



7. An AES spectrum from the fracture surface of a 7050-T73 Al alloy. Notice the presence of Pb on the grain boundaries.

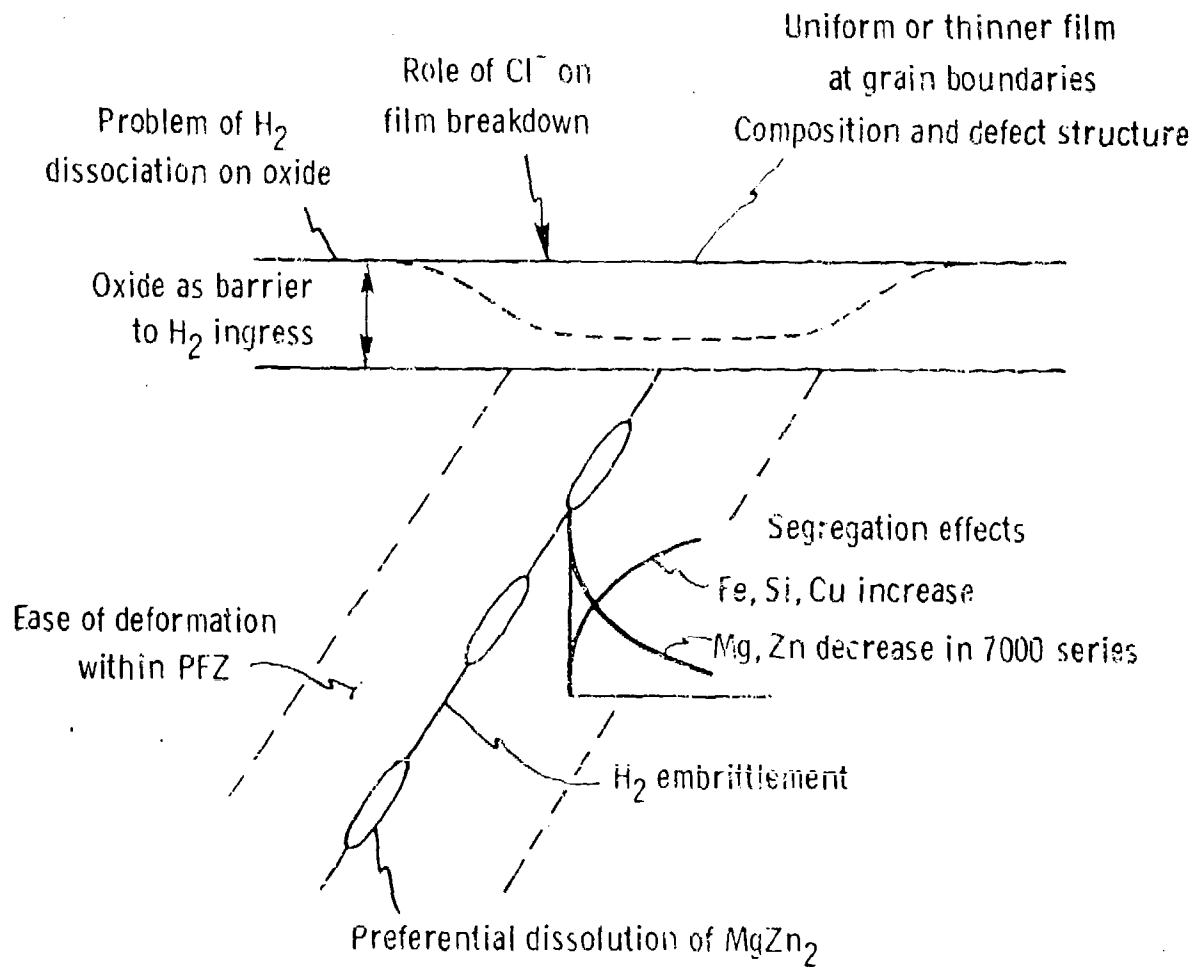


8. Chemical depth profiles of 7050 Al alloy in T73 and T76 tempers.



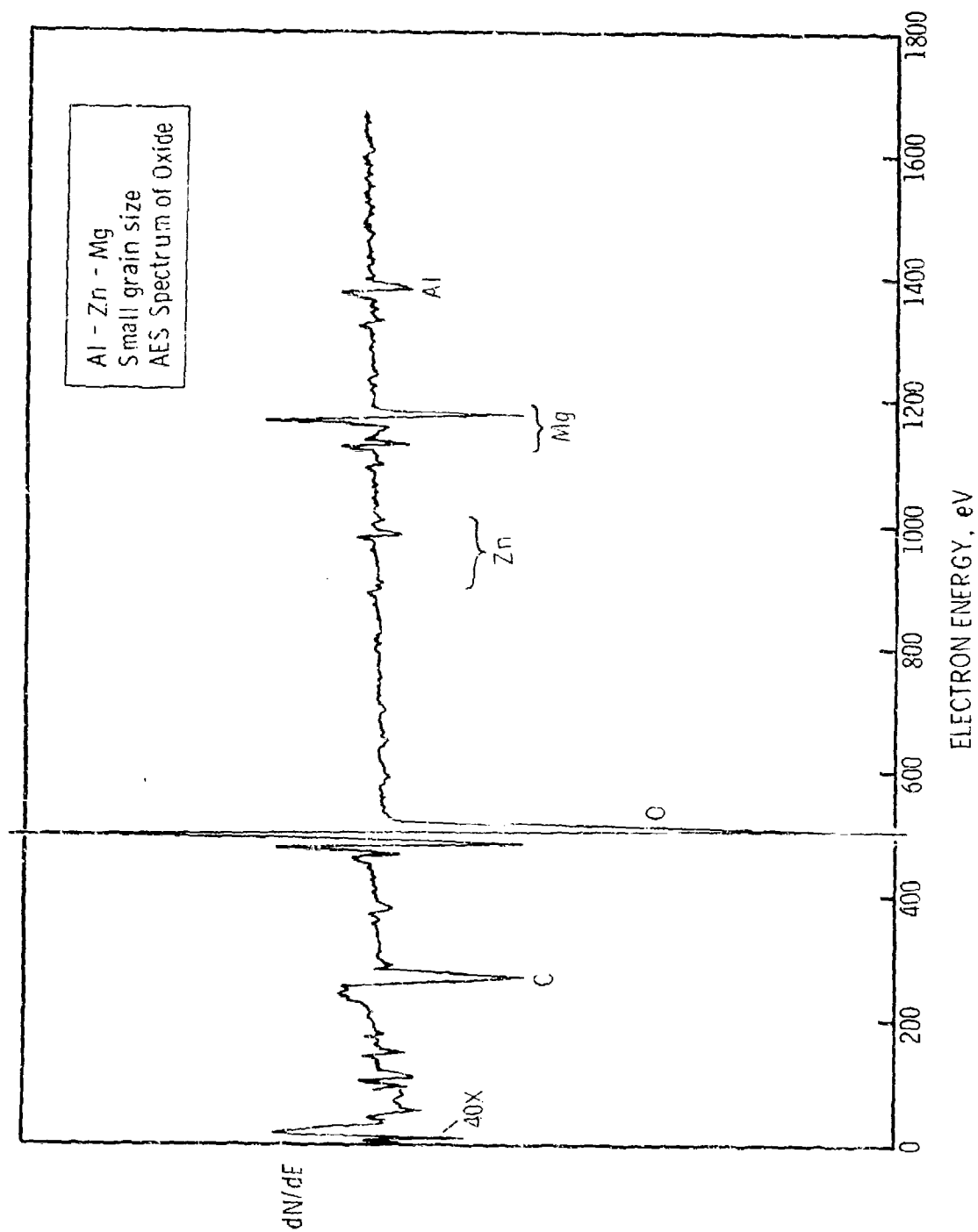
9. Chemical depth profiles of 7049 Al alloy in T73 and T87 tempers.

SCHEMATIC  
ROLE OF OXIDE FILMS IN  
STRESS CORROSION CRACKING OF ALUMINUM ALLOYS



10. A schematic diagram of phenomena and processes associated with the initiation and propagation of stress corrosion cracks in high strength Al alloys.





11. An AES spectrum of the oxide on a solution-treated and quenched Al-Zn-Mg high purity ternary alloy with small grain size ( $\sim 0.08$  mm).

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